

REMARKS

Reconsideration and withdrawal of the Examiner's rejections under 35 USC §102(e) and double patenting is requested in view of the foregoing amendments and the following remarks.

35 USC §102

The examiner has rejected claims 10-11 under 35 U.S.C. 102(e) as being anticipated by Leupin, et al., U.S. Patent No. 6,384,011; asserting that Leupin, et al., U.S. Patent No. 6,384,011, discloses a laundry detergent composition comprising 0.1-5% by weight of a hydrophobically modified cellulose material (see col. 4, line 26-col. 5, line 65), 1-80% by weight of a deterative surfactant, such as a combination of anionic and nonionic surfactants (see col. 6, lines 7-65), 0.1-80% by weight of a builder, such as silicates and aluminosilicates (see col. 7, line 56-col. 8, line 30), perfumes (see col. 8, lines 31-43), and 5-12% by weight of water (see col. 12, lines 25-32), per the requirements of the instant claims; that it is further taught by Leupin et al that the composition is used in a method to treat fabrics to impart fabric appearance benefits (see col. 12, lines 59-67); Specifically, note Examples 1-6; and, therefore, instant claims 10-11 are anticipated by Leupin, et al., U.S. Patent No. 6,384,011. In response, applicants have amended claims 1 and 6 and cancelled claims 2, 7 and 11 to clearly distinguish the instant invention over Leupin, et al.

Leupin, et al., discloses cellulosic based polymers and oligomers of the general formula:

beta 1-4 D-glucan backbone modified with R groups, where $R = H$ or R_c , and where $R_c = -(CH_2)_yCOOZ$. Here Z can stand for a range of different groups but the list does not include pendant polysaccharide or saccharide groups. If $R = H$ then the resulting polymer is cellulose. If $R = R_c$ then the resulting polymers are modified sodium carboxymethyl celluloses and the list given for Z does not include pendant polysaccharide chains or saccharide groups

Claims 1, 6 and 10 have been amended by adding the limitations of Claims 2, 7 and 11, respectively – i.e., pendant polysaccharides selected from galactomannan, glucomannan, xyloglucan, xanthan gum and mixtures thereof. All of these have different structures from the structures disclosed or suggested in Leupin, et al., (US 6384011B1) since they all have pendant polysaccharide or saccharide groups and none are based on the carboxymethyl cellulose structure. The galactomannan and glucomannan polysaccharides also have different backbone chains which contain 2 sugar units (i.e. not based on the 1-4 D-glucan backbone as is the case with Leupin). Galactomannan consists of a backbone of beta-1,4-glycosidically linked mannopyranosyl residues with a-D-galactopyranosyl residues linked to the O-6 of certain mannose moieties. (Industrial Polymers Handbook, Volume 4, Edited by Edward S. Wilks, published by Wiley-VCH, p. 2085; attached). Glucomannan contains both glucose and mannose in the backbone chain. (Cellulose chemistry and its applications, editors T.P. Nevell

and S.H. Zeronian, published by Ellis Horwood Limited, p38; attached). Xyloglucan has a glucan backbone with xylose or xylose + galactose & fucose pendant chains (Cellulose chemistry and its applications, editors T.P. Nevell and S.H. Zeronian, published by Ellis Horwood Limited, p38; attached). Xanthan Gum is a β -1,4-glucan backbone with trisaccharide of glucose, glucuronate & carboxymethyl mannose linked to C3 on alternative residues of the backbone (Cellulose chemistry and its applications, editors T.P. Nevell and S.H. Zeronian, published by Ellis Horwood Limited p105-106; attached).

Therefore, the polysaccharides of amended claims 1, 6 and 10 are fundamentally different from those disclosed or suggested by Leupin.

Provisional Obviousness-Type Double Patenting Rejection

The examiner has maintained the rejection of claims 1-7 and rejected claims 10-11 under the judicially created doctrine of obviousness-type double patenting as being unpatentable over claims 1-17 of copending Application No. 10/239,967; over claims 1-21 of copending Application No. 10/225,863; and over claims 1-31 of copending Application No. 10/225,864.

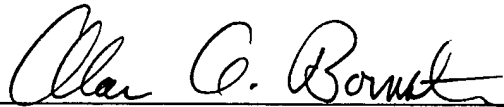
The examiner notes that applicant has not provided terminal disclaimers over copending Application Nos. 10/239,967, 10/225,863 and 10/225,864. It is noted by the examiner that these applications have not issued as U.S. patents, and thus maintains that the instant claims are still provisionally

rejected over these applications. Applicants further note that the examiner agrees with applicant in that if these copending applications do not issue as U.S. patents at the time allowable subject matter is found in the instant application, that these provisional rejections will be removed, per the requirements of MPEP 804.

CONCLUSION

In summary by the present amendments, claims 1, 6 and 10 have been amended and claims 2, 7 and 11 have been cancelled. Applicants submit that no new matter has been added by these amendments. In light of the above amendments and remarks, applicants submit that all claims now pending in the present application are in condition for allowance. Reconsideration and allowance of the application is respectfully requested. The telephone interview would facilitate prosecution of the application, the examiner is invited to contact the undersigned.

Respectfully submitted,

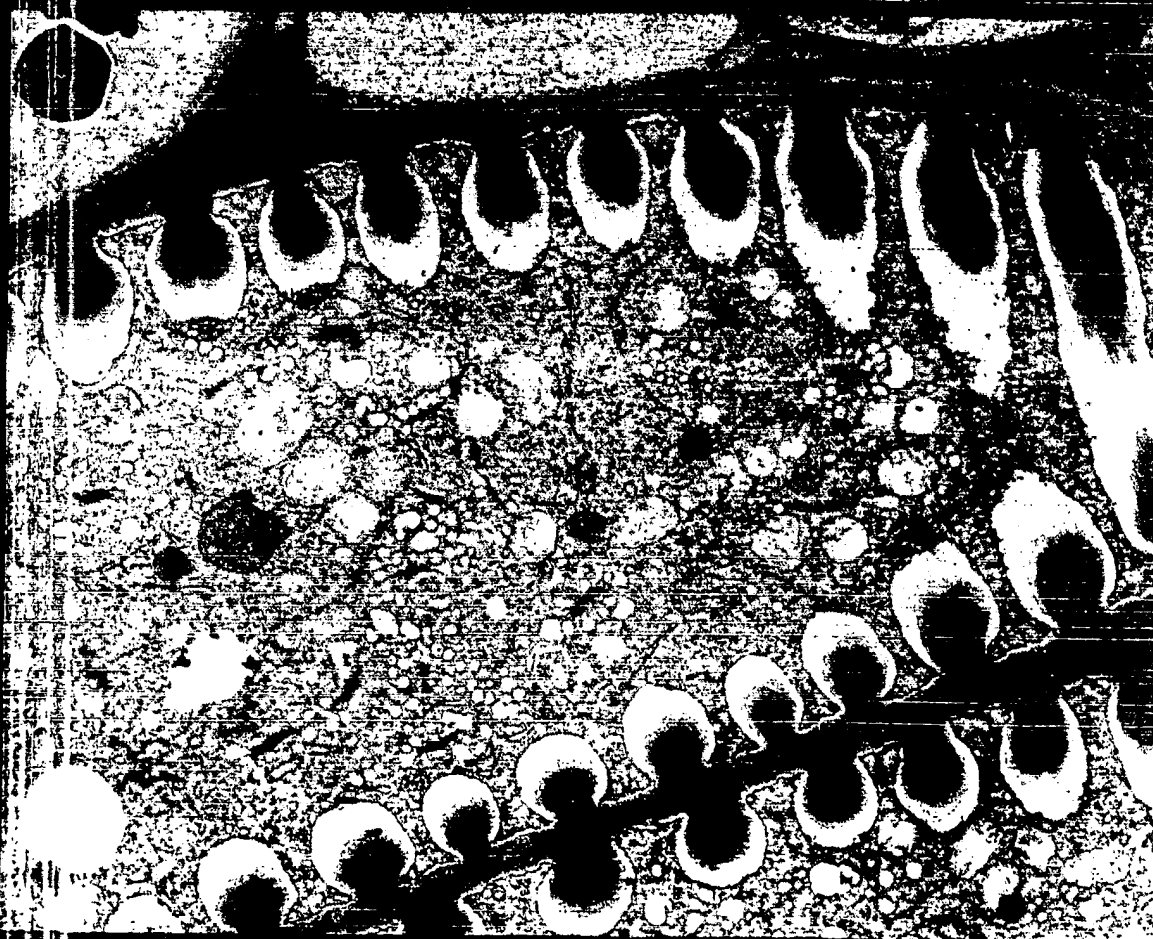
A handwritten signature in black ink, appearing to read "Alan A. Bornstein", written over a horizontal line.

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AAB/ss
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Cellulose Chemistry and its applications



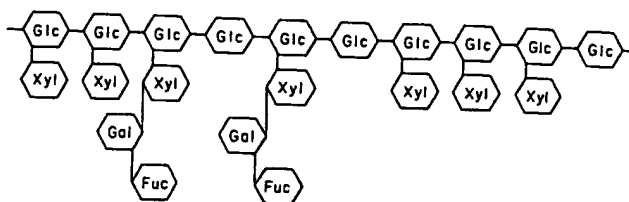


Fig. 2.2 - Xyloglucan, a typical hemicellulose molecule, has a linear backbone of glucose (Glc) residues with side branches composed of a single xylose (Xyl) residue or xylose plus galactose (Gal) and fucose (Fuc). (Redrawn from *The Molecular Biology of the Cell*, 1983, Garland, New York.)

both xylosyl and glucosyl residues in the main chain and unbranched xylans have been isolated [33,88].

The secondary walls of gymnosperms contain large amounts of hemicellulosic glucomannans (DP of 100–400) and minor amounts of arabino-4-*O*-methylglucurono-xylan [77,78,84]. The two types of glucomannan commonly present are *O*-acetyl galactoglucomannan and an unacetylated galactoglucomannan (ratios Gal:Glc:Man of 1:1:3 and 0.1:1:4, respectively). Both of these molecules contain both glucose and mannose in the main chain [77,78,86,88].

The main hemicellulose extracted from dicot primary cell walls is xyloglucan (Fig. 2.2). Walls from suspension-cultured cells and intact plants contain about 19% xyloglucan (DP of about 50) and 5% glucuronoarabinoxylan [33]. Minor amounts (about 2%) of xyloglucan are also present in monocot primary cell walls where arabinoxylan is the main hemicellulose [33].

In contrast to the marked decrease in pectin deposition during secondary wall formation, the deposition of hemicelluloses continues during cell maturation [87]. Dicot primary walls contain about 24% hemicellulose and mature walls in wood contain 15–35% hemicellulose [77,78]. The proportion of hemicellulosic polysaccharides has been shown to increase in secondary walls of several higher plants [20]. The continued deposition of hemicelluloses is consistent with their proposed function in hydrogen bonding to cellulose [33], which increases in percentage in secondary walls. Hemicelluloses form aggregates in solution [89,90], suggesting that they may also participate in gel formation within the wall.

2.3.5 Protein

The primary walls of all higher plants contain 5–10% protein; secondary walls contain only negligible amounts of protein [32]. The major protein isolated from dicot primary walls is a glycoprotein containing 20% of the rare amino acid, hydroxyproline, and covalently bound arabinosyl and galactosyl residues. Structural analysis has shown that the pentapeptide, serine-(hydroxyproline)₄,

ending as cellulose I is proposed; cellulose III lattice. Model chains II₁ lattice and tested against the proposes that the chain conformation as in cellulose II. *R* factors for the same.

pattern called cellulose IV. It also s, as in the case of cellulose III, ng on their source. The lattice ogen bonding and chain packing ne as in parallel models of cellulose is similar to antiparallel models termination of cellulose IV.

he interaction of cellulose and use they constitute intermediates original work was summarized by

t complex structures were found t of NaOH present. The nomen- and Okano attempted to clarify of the structures of the soda e have been defined. Na cellulose OH and the usual two-fold chain a three-fold chain conformation it too is an almost fully extended ated that there might be a four-nixed samples with some crystal-three-fold helices. A pattern from fold structures would have layer

s no Na [77]. A justification for penultimate structure along the results from the treatment with also and Okano preferred to not cellulose hydrate' had historically (generated) even though no been reported for cellulose II ose-N₂H₄ complex with the same. Both are the

same as the historic 'water cellulose'. The polarity of chains and their conformation in cellulose II hydrate are similar to that proposed for cellulose II. The main difference in the two structures is the presence of interstitial water molecules.

Some work has been reported on the three-fold structure of Na cellulose IIb [80], but it was not determined whether the chains were parallel or antiparallel.

Sarko *et al.* [81] have proposed that the conversion from parallel to antiparallel chains occurs during the formation of the mixed cellulose I - Na cellulose I crystals, the first step in the conversion. Because of the additional material in the unit cell, a sufficient quantity and quality of data to settle the chain polarity question may not be forthcoming from the soda celluloses. However, a preliminary report [81] indicates that Na cellulose IIb is antiparallel.

Although the Na celluloses I and III are two-fold helices, their fiber repeat is shortened slightly, from about 1.034 nm to 1.012 nm.

The effects of NaOH on cellulose are only partly indicated by the formation of the soda celluloses. Further documentation of the effects of variables in the treatment, such as temperature, physical restraint of the fiber from shrinkage, and concentration is needed before the process is understood.

3.4.6 Cellulose complexes

In addition to cellulose hydrate [77,79], recent structural work is available on cellulose I - ethylenediamine [82] and cellulose II - hydrazine [83]. In both, the chain conformation was similar to the parent structure except for the O6 conformation in the ethylenediamine structure. There, the O6 atoms are oriented *gt*. The chain packing is different, however, with the chains in register rather than staggered by 1/4 of the *z*-axis. The complexing agent is placed between stacks of chains, disrupting the hydrogen-bonded sheets of native cellulose.

Some ideas as to the extent of variety of amine complexes possible can be gained from the papers by Creely *et al.* [84,85] and the references therein.

3.4.7 Cellulose derivatives

When there are more atoms in the unit cell, more data are required for a structural determination comparable in quality to those of simpler structures. At the same time, the substituents added to the cellulose chain usually are attached to the O2, O3 or O6 atoms and should have substantial freedom of rotation, which should lead to poorer crystallization. However, some fine X-ray patterns have been made and full determinations of structure have been reported for cellulose triacetate I [86] and II [87]. The acetate substituents take a predictable orientation with regard to the glucose residue. The CTA I structure is reported to consist of parallel chains and CTA II of antiparallel chains.

The cellulose nitrate structure [88] is a helix with five residues in two turns, also being almost fully extended.

Because of its β -1,4-glucan backbone, xanthan could be considered a natural derivative of cellulose. The side chains consist of a trisaccharide of glucose,

glucuronate and carboxyethyl mannose, linked to the C3 on alternate residues of the backbone. Its structural study has been difficult, with a reversal of the conclusion as to whether xanthan molecules form single or double helices [89, 90]. Solution characteristics have been important in its current characterization as a double helical structure [91].

3.5 SMALL ANGLE X-RAY SCATTERING (SAXS)

SAXS can be used to detect periodicities of a larger scale than those found with the more 'normal' wide angle diffraction studies. The major experimental difference is an increase in the distance between the sample and the film or recording device. The periodicities correspond to the crystallite length and width. Although X-rays are the usual radiation, small angle neutron diffraction has also been used [92].

SAXS results can be correlated successfully with the interpretations of peak widths measured at half-height. A more helpful use for SAXS is to determine the number and size of pores within fibers [93,94].

For these measurements, the scattering due to spacings between 20 and 100 nm is recorded. The scattering is more diffuse than at larger angles (smaller spacings), and the calculation of the relative number of pores is related to the shape of the intensity curve. The importance of this work for practical purposes is not to be underestimated, especially for rayon fibers, which vary greatly in the number and size of pores. These variations in properties result from changes in the manufacturing processes and have substantial impact on mechanical performance and on dye uptake, to give two examples.

3.6 SPECTRAL STUDIES OF CELLULOSE AND RELATED COMPOUNDS

Infrared, Raman, and recently, ^{13}C nmr magic angle spinning spectroscopy have been used to study the conformation of cellulose. The early work with infrared showed spectral differences between the celluloses from algae and bacteria on the one hand and from cotton and ramie on the other [95]. Polarized infrared studies identified the orientation of hydrogen bonds within the crystalline regions [96]. Early work also established differences between native cellulose and the other allomorphs, and an infrared determination of crystallinity has been used.

Very highly resolved infrared and Raman spectra are possible, with differences between different preparations of ostensibly the same material being easily detectable. A thorough analysis of the vibrational spectra for cellulose is summarized by Blackwell [97]. It was concluded that the vibrational modes are highly complex and much further work is needed before the spectra can be fully understood.

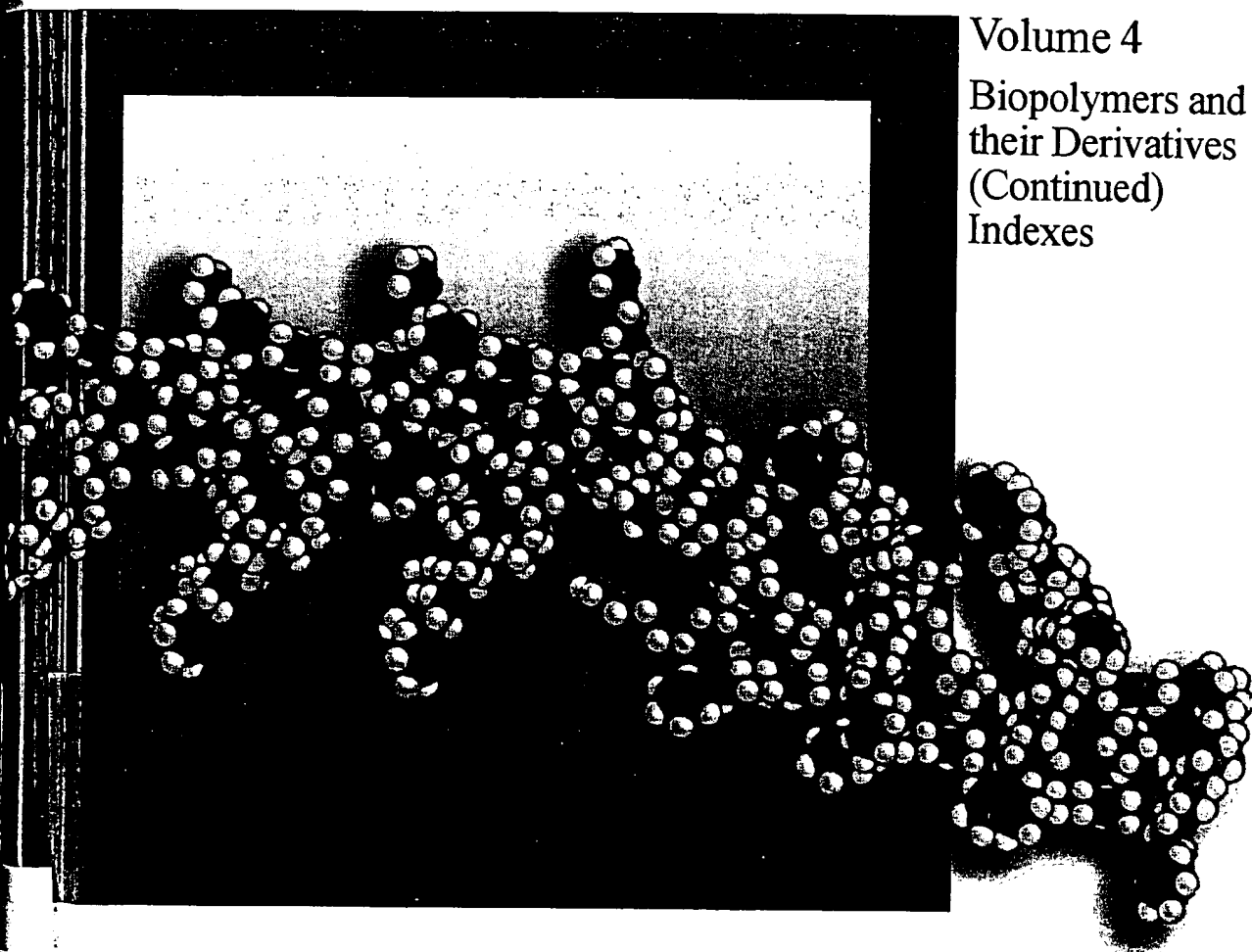
Industrial Polymers Handbook

Products, Processes, Applications

Edited by Edward S. Wilks

Volume 4

Biopolymers and
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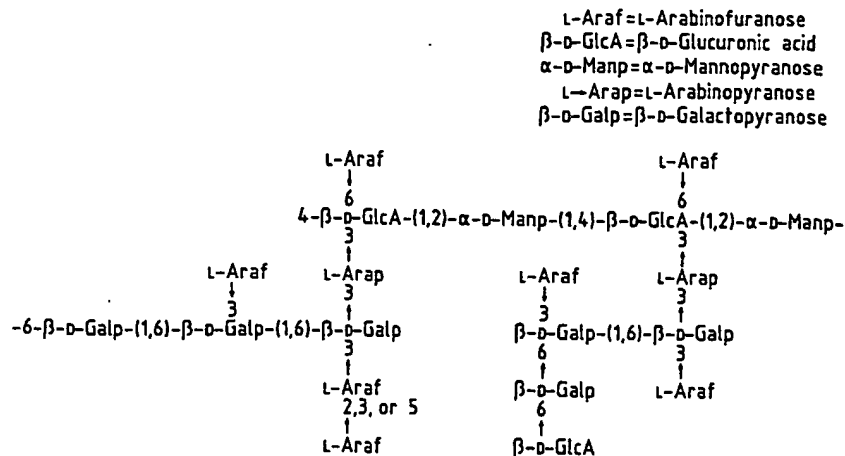


Figure 22. Structural features of gum ghatti

9.11. Xanthan Gum

Xanthan gum is the extracellular, high molecular mass heteropolysaccharide produced by the aerobic fermentation of *Xanthomonas campestris* NRRL-B1459 [1]–[5], [222]. The production of xanthan gum goes back to the investigations of JEANES and coworkers in the early 1960s at the Northern Regional Research Laboratory of the United States Department of Agriculture, Peoria, Illinois, in the framework of a large screening program for slime-producing microorganisms of industrial interest [223].

9.11.1. Production

Commercial production is carried out by a batchwise, submersed fermentation under strong aeration. During fermentation the bacterial cells are kept under a constant stress to direct their metabolism to metabolite production instead of growth. This is achieved by proper selection of medium composition. The medium cost forms a significant part (ca. 25 %) of the total production cost; the sugar concentration is adjusted so that it falls as low as possible at the end of the fermentation cycle, but depletion must be avoided to prevent the microorganisms from using the polysaccharide as a substrate [224]. For xanthan gum production, a typical medium contains a nitrogen source, phosphate and magnesium ions, trace elements, and glucose, which is kept at < 5 %. The pH must not be below 7. After 96 h at 30 °C, less than 0.1 % glucose is left in the medium, and a conversion to polysaccharide of more than 50 % can be obtained.

In the patent literature [225], much attention is devoted to propagation of the starter culture. In this stage, nitrogen is supplied by organic sources (e.g., soy peptone or corn

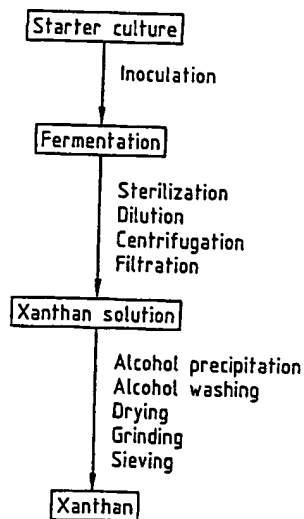


Figure 23. Flow sheet for the manufacture of xanthan gum

steep liquor), which also contain some growth factors. During the main fermentation, nitrogen is supplied by mineral salts (e.g., ammonium nitrate). A typical composition of the medium at this stage is 0.06 % ammonium nitrate, 0.5 % potassium dihydrogenphosphate, 0.01 % magnesium sulfate heptahydrate, 2.25 % glucose, and 97.18 % water (Fig. 23). After inoculation of 5300 L of medium with 227 L of starter culture and vigorous aeration for 72 h at 28–31 °C, 77 kg of hydrocolloid can be recovered. The remaining medium contains < 0.1 % glucose. This means a conversion of 64.7 % and a final concentration of the polysaccharide of 1.45 %, which gives the medium a viscosity of 3000 mPa · s at 25 °C (Brookfield Viscometer, 60 rpm).

The usual precautions must be taken to preclude contamination of the batch fermentation through the culture medium, equipment and accessories, air, and neutralizer; thus it is doubtful if the batch operation can be replaced by a continuous process. Also the stability of the bacterial strain is a point of consideration. Since *Xanthomonas* bacteria do not form spores, the fermentation can be stopped by heat treatment in a heat exchanger. Further downstream processing involves centrifugation, filtration, and precipitation with ethanol or isopropanol as used for other hydrocolloids. The high viscosity of the fermentation liquor must be reduced by dilution with water or alcohol. The precipitate, dried by vacuum or with hot air, is processed to a marketable article by grinding and sieving. The powder is off-white to yellow in color.

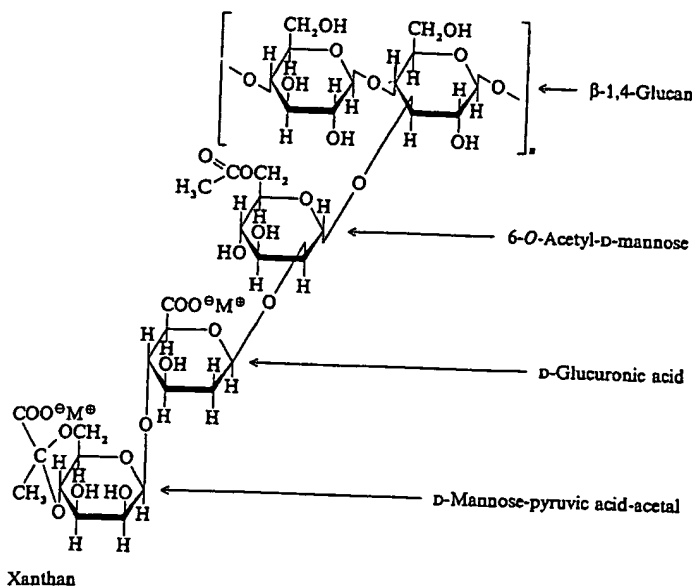


Figure 24. Structural features of xanthan gum

9.11.2. Structure and Properties

Chemical Structure. Xanthan is a heteropolysaccharide composed of D-glucose, D-mannose, and D-glucuronic acid. It has a β -1,4-D-glucan (cellulosic) backbone substituted through C-3 on alternate glucose residues with a trisaccharide side chain consisting of β -D-mannose-(1,4)- β -D-glucuronic acid-(1,2)- α -D-mannose. The terminal mannose moiety may have pyruvate residues linked to the 4- and 6-positions, the internal mannose unit is acetylated at C-6 (Fig. 24) [226]. X-ray diffraction studies on oriented xanthan gum fibers [227] identified the molecular conformation as a right-handed five-fold helix with a pitch of 4.7 nm. In this conformation the trisaccharide side chain is aligned with the backbone and stabilizes the overall conformation by hydrogen bonding [228].

By modifying the biosynthetic pathway for xanthan production, the carbohydrate structure and the substitution pattern of the polymer can be genetically controlled. In this way, polysaccharides with quite different properties can be obtained [229].

Xanthan gum has a *molecular mass* of ca. 2×10^6 . Since the structure and the molecular mass are genetically controlled, hydrocolloids with similar molecular mass and similar properties are obtained.

Solution Properties. Xanthan gum is completely soluble in hot or cold water. The gum can be hydrated in sugar solutions up to 60 %. The viscosity of the gum solution is a function of its concentration in the dispersion [230]. *Aqueous solutions* of xanthan gum are extremely viscous (Table 4) and pseudoplastic (i.e., they exhibit a reversible, highly

Hong Kong, Philippines, Taiwan and Thailand, most of South America, and all 15 EU member states.

In the European Union gellan gum is one of the generally permitted additives listed (as E418) in Appendix I of the European Parliament and Council Directive 95/1/EU on food additives other than colors and sweeteners [278].

Specification and Purity. Kelcogel gellan gum complies with the specifications for gellan gum prepared by the Joint FAO/WHO Expert Committee on Food Additives (JECFA) [279] and the Food Chemicals Codex (FCC) [280].

Market. The commercially available products are K9A50, a nonclarified form of the gum for industrial applications; Kelcogel LT100, a nonclarified form and Kelcogel, a clarified form for food and industrial uses; and Gelrite, which is used for microbiological plating media. All gellan gum is produced by NutraSweet Kelco (a Monsanto company). Gellan gum is priced at about \$ 53 per kg.

9.13. Galactomannans [2]–[6], [11], [281]

Galactomannans are plant reserve carbohydrates, like starch, that occur in the endosperm of the seeds of many Leguminosae. During sprouting, the galactomannans are enzymatically degraded and used as nutrition. Of the many known galactomannans, up to now only three have been processed and used on an industrial scale:

- 1) **Locust bean gum (carob gum)** has been known for a long time. It is derived from seeds of the carob tree (*Ceratonia siliqua* L.) which grows around the Mediterranean Sea, in Spain, Portugal, and Morocco, where more attention is paid to its systematic cultivation. The annual production is estimated at 10 000–12 000 t.
- 2) **Guar gum** is at present the most important galactomannan. It is derived from the seeds of the guar plant (*Cyamopsis tetragonoloba* L. Taub.), which grows in India and Pakistan. Since 1944, guar plants have also been cultivated on a large scale in the southern United States. Recently, they have been introduced into Brazil, Africa, and Australia, where two crops a year can be obtained. The industrial production of guar gum began around 1940. The annual production is estimated at ca. 125 000 t.
- 3) **Tara gum** has been produced only since the 1970s, to a much lesser degree (1000 t/a), from the seeds of the tara tree (*Cesalpina spinosa* L.), which occurs mainly in Peru.

The galactomannans on the market are flours prepared from the endosperm of the corresponding seeds. The carob and tara kernels are particularly difficult to process because of their tough, hard hulls. Remnants of the hulls and seeds are, therefore, always present in small amounts. The pure polysaccharide extracted from the flour of

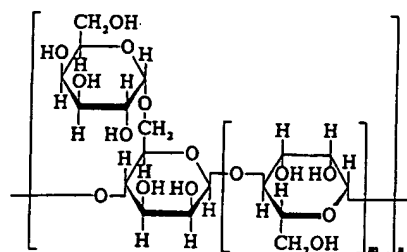


Figure 26. Structural features of galactomannans

$m = 3$: Locust bean gum
 $m = 1$: Guar gum
 $m = 2$: Tara gum

locust bean gum is often designated carobin gum and the polysaccharide from guar flour, guaran.

9.13.1. Structure

Galactomannans have a backbone of $\beta(1,4)$ -glycosidically linked mannopyranosyl residues. This backbone is substituted with single-unit α -D-galactopyranosyl residues linked to the O-6 of certain mannose moieties. This substitution renders the mannans soluble. Within the various mannans a wide spectrum of chemical structures occurs. This diversity of structure includes not only a wide variation in their mannose: galactose ratio, but also significant differences in the distribution of galactose units along the mannan backbone (Fig. 26) [282], [283]. For *locust bean gum* a mannose: galactose ratio of 3.5 has been established, compared to a ratio of 3 for *tara gum* and 1.5 for *guar gum*. A measure of the differences in the distribution of galactose side groups has been obtained from an examination of the degree of hydrolysis and the characteristic array of oligosaccharides obtained by digestion with an endomannanase. In this way, DEA et al. [284] established that a *tara gum* has a more statistically random distribution of side chains while *locust bean gum* has a nonregular, nonstatistically random distribution with a certain proportion of unsubstituted blocks of intermediate length. *Guar gum* was found to have few, if any, regions that were unsubstituted (smooth regions) with galactose. Galactomannans differ further in their molar mass distribution; the highest values were established for guar gum. These differences in fine structure between the galactomannans, involving the extent and pattern of galactosyl substitution and molar mass distribution, account for their very different functional properties.

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